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<b>(21) International Application Number:</b> PCT/EP99/01563 <b>(22) International Filing Date:</b> 8 March 1999 (08.03.99) <b>(30) Priority Data:</b> 9806714.3 27 March 1998 (27.03.98) GB <b>(71) Applicant (for AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW only):</b> UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). <b>(71) Applicant (for all designated States except AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT UG US ZW):</b> UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). <b>(71) Applicant (for IN only):</b> HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Mumbai 400 020, Maharashtra (IN). <b>(72) Inventor; and</b> <b>(75) Inventor/Applicant (for US only):</b> KORB, Shimei [GB/GB]; Unilever Research Port Sunlight, Quarry Road East, Bebington, Wirral, Merseyside L63 3JW (GB).		<b>(74) Agent:</b> MOLE, Peter, Geoffrey; Unilever plc, Patent Dept., Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). <b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> FABRIC SOFTENING COMPOSITION  <b>(57) Abstract</b>  A fabric softening composition comprising 1 to 8 % by weight of one or more quaternary ammonium fabric conditioning compounds, stabilising agent and fatty alcohol having low and high temperature stability.		

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FABRIC SOFTENING COMPOSITION

The present invention relates to Fabric Softening compositions, in particular the invention relates to aqueous  
5 dispersions of biodegradable fabric softening compositions comprising a water insoluble cationic fabric softening agent, a stabilising agent and a fatty alcohol suitable as a rinse-added fabric softener composition.

10 Rinse added fabric softener compositions are well known. Typically such compositions contain a water insoluble quaternary ammonium fabric softening agent dispersed in water at a level of softening agent up to 8% by weight in which case the compositions are considered dilute, or at  
15 levels from 8% to 50% in which case the compositions are considered concentrates. In addition to softening, fabric softening compositions desirably have other benefits. One is the ability to confer soil release properties to fabrics, particularly those woven from polyester fibre. Other  
20 benefits include improved perfume delivery.

One of the problems associated with fabric softening compositions is the physical instability of such compositions when stored.

25

This problem is particularly noticeable in storage of dilute compositions at variable temperatures. Such composition tend to become unstable after storage at variable temperatures over time. Consumers, however, have high

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expectations of characteristics such as viscosity and pourability from fabric softening compositions, expecting a specific level of viscosity and ease of pour which makes the composition convenient as well as pleasurable to use.

- 5 Physical instability of dilute compositions manifests as a decrease in viscosity to a level which is unacceptable, particularly in view of the consumers high demands. Furthermore, unacceptable levels of thickening/gelling and phase separation can also occur as a result of storage over  
10 time and particularly at highly variable temperatures.

- In the past physical stability of rinse added fabric softener compositions has been improved by the addition of viscosity control agents or anti-gelling agents. In EP  
15 280550 (Unilever) it has been proposed to improve the physical stability of dilute compositions comprising biodegradable, ester-linked quaternary ammonium compounds and fatty acid by the addition of nonionic surfactants.

- 20 Certain stabilising agents not only stabilise fabric softener composition comprising biodegradable quaternary ammonium compounds but are also environmentally friendly, in that they show acceptable biodegradability and are not substantially toxic in aquatic systems.

25

It is also known from EP 13780 (Procter & Gamble) to form concentrated aqueous liquid fabric softening compositions from a mixture of a water-insoluble, cationic material and a nonionic material selected from hydrocarbons, fatty acids,

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fatty esters and fatty alcohols; the nonionic material acting to improve the viscosity characteristics of a product when the level of cationic material is above 8%.

- 5 It is also known from GB 2039556 and EP 13780 that preferred compositions can also include low levels of electrolyte such as respectively sodium chloride or calcium chloride, to further control product viscosity.
- 10 EP 056 695 (Procter & Gamble) discloses compositions comprising 12.3-25% cationic fabric softener and ethoxylated amines. WO 92/15745 (Procter & Gamble) discloses compositions comprising 10-35% cationic fabric softener, a highly branched fatty alcohol and a fatty alcohol
- 15 ethoxylate.

- GB 2 170 829 and EP 394 133 (Colgate-Palmolive) disclose compositions comprising 3-8% and 2-35% respectively cationic fabric softener that do not need non ionic surfactant
- 20 emulsions to keep viscosity and stability acceptable.

- GB 2 139 259 and EP 122 141 (Unilever) disclose compositions comprising 8% or more cationic fabric softener and non ionic materials with an HLB of less than 10, e.g. fatty alcohols.
- 25

EP 409 504 (Unilever) disclose compositions comprising cationic fabric softener and fatty alcohols as extenders for the softener.

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GB 1 538 094 discloses compositions comprising cationic fabric softeners, fatty alcohols and non-ionic emulsifiers.

Surprisingly, we have found that low and high temperature  
5 stability of dilute compositions containing biodegradable quaternary ammonium compounds may be improved by the addition of a mixture of selected stabilising agents and fatty alcohol.

10 Thus, according to one aspect of the invention there is provided a fabric softening composition comprising:

- a) from 1 to 8% by weight of one or more quaternary ammonium fabric conditioning compounds;
- 15 b) a stabilising agent selected from a nonionic surfactant or a single long chain alkyl cationic surfactant or mixtures thereof ;and
- c) a fatty alcohol

20 Such compositions show improved high temperature stability, (in particular stability at 37°C), stability at ambient temperatures as well as stability at low temperatures. By high temperature stability is meant stability at temperatures above the ambient temperature to which the  
25 composition is normally exposed. Low temperature stability means stability at temperatures below the ambient temperature to which the composition is normally exposed.

- 5 -

According to another aspect of the invention there is provided a process, for making a liquid fabric softening composition of the invention, comprising the steps of;

- 5 (i) mixing and heating the quaternary ammonium compound, stabilising agent and fatty alcohol to form a melt and
- (ii) dispersing the melt in water.

- 10 According to another aspect of the invention there is provided the use of a composition according to the invention to form a stable aqueous fabric softening composition.

- According to another aspect of the invention there is
- 15 provided the use, in a fabric softening composition comprising a water insoluble quaternary ammonium material, of a mixture of a stabilising agent and fatty alcohol, in order to improve the low, ambient and high temperature stability of the composition.

- 20 Preferably, the compositions of the invention are liquids comprising an aqueous base.

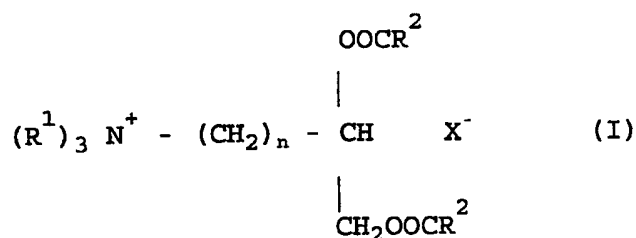
- At least one of the one or more quaternary ammonium fabric
- 25 conditioning compounds is in, one preferred embodiment, a quaternary ammonium compound having at least one ester linkage, especially a compound having two C<sub>8-20</sub> alkyl or alkenyl groups connected to the quaternary ammonium head group via at least one ester link. It is more preferred if

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the quaternary ammonium compound has two ester links present. Preferably the fabric softening composition contains either one or more ester linked quaternary ammonium compounds, or, one or more non-ester linked quaternary ammonium compounds.

Preferably the quaternary ammonium fabric conditioning compounds for use in the compositions according to the invention are represented by the formula:

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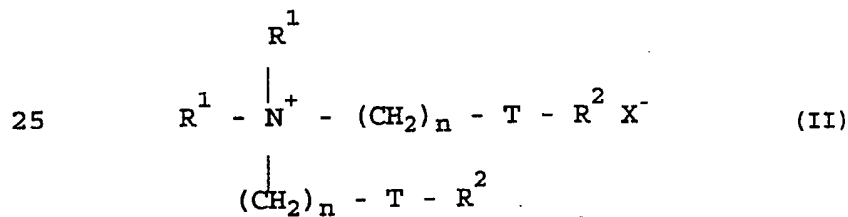


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wherein each  $\text{R}^1$  group is independently selected from  $\text{C}_{1-4}$  alkyl, alkenyl or hydroxyalkyl groups; each  $\text{R}^2$  group is independently selected from  $\text{C}_{8-28}$  alkyl or alkenyl groups;  $\text{X}^-$  is a compatible anion; and  $n$  is an integer from 0-5;

20

or by the formula:

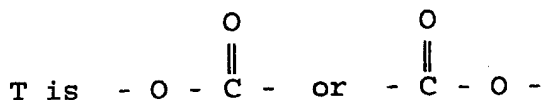


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wherein  $\text{R}^1$ ,  $\text{X}^-$ ,  $n$  and  $\text{R}^2$  are as defined above, and



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5 The preferred compounds of type (I) and their method of preparation are, for example, described in US 4 137 180 (Lever Brothers). A particularly preferred compound of type (I) is 1,2 bis[hardened tallowoyloxy]-3-trimethylammonium propane. Preferably these compounds comprise small amounts  
 10 of the corresponding monoester as described in US 4 137 180 for example 1-hardened tallowoxy 2-hydroxy-3-trimethylammonium propane chloride.

The preferred compounds of type (II) is di[2-(hardened  
 15 tallowoyloxy)ethyl] dimethylammonium chloride.

Of the quaternary ammonium compounds not having an ester linkage suitable examples include di-(hardened tallow) dimethylammonium chloride and the compound sold under the  
 20 Tradename 'Accosoft 460HC'.

The level of quaternary ammonium compound is from 1% to 8% by weight of the total composition, more preferably from 1.5% to 7.9%, most preferably from 2% and 7%, such as 3% to  
 25 5%.

In a preferred embodiment of the invention, the ratio of stabilising agent to fatty alcohol is greater than or equal to 1:1. This range provides improved physical stability of  
 30 the composition. The weight ratio of stabilising agent to

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fatty alcohol is preferably is greater than or equal to 1:1.5, more preferably greater than or equal to 1:1.8. Higher ratios of greater than or equal to 1:3 or 1:7 are also effective. Depending upon the required viscosity of the product ratios of greater than or equal to 1:10 may be used. By adjusting the ratio above 1:1 the desired viscosity of the product is obtained. Typically the higher the proportion of fatty alcohol used the higher is the resultant viscosity of the product.

10

Compositions comprising from 1 to 8% level of quaternary ammonium compound are considered dilute fabric softening compositions. These pose a different problem in managing product viscosity via stability of the composition than the problem posed by concentrated fabric softening compositions.

15

Compositions according to the invention contain a stabilising agent selected from a nonionic surfactant or a single long chain alkyl cationic surfactant or mixtures thereof.

20

Suitable nonionic surfactant stabilisers which can be used include the condensation products of  $C_8$ - $C_{30}$  primary or secondary, linear or branched alcohols alkoxylated with 10 or more moles of alkylene oxide, preferably 10 to 20 moles of alkylene oxide, more preferably between 15 and 20 moles of alkylene oxide. Preferably the condensation product is of a  $C_{10}$ - $C_{22}$  primary or secondary, linear or branched alcohol.

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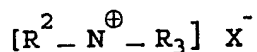
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Preferably the alkylene oxide is ethylene oxide. The alcohols may be saturated or unsaturated. Particularly preferred are Genapol T-110, Genapol T-150, Genapol T-200, Genapol C-200, Genapol 0-100 and Genapol 0-150 all ex  
5 Hoechst AG, Lutensol AT18 ex BASF. Preferably the nonionic stabiliser has an HLB of between 10 and 20, more preferably 12 and 20.

Preferably, the level of nonionic surfactant stabiliser is  
10 within the range of from 0.01 to 10% by weight, more preferably from 0.05 to 5% by weight most preferably from 0.1 to 3% by weight, such as 0.15 to 1.5% by weight. Frequently amounts of 0.01 to 1% by weight, such as 0.01 to 0.5% of nonionic surfactant stabiliser may be used.

15 Further possible stabilisers suitable in the present invention are mono (single) long chain alkyl cationic surfactants. Preferably, quaternary ammonium salts of the general formula:

20



25 wherein the  $R^2$  group is  $C_{10}$ - $C_{22}$  hydrocarbon group, preferably  $C_{12}$ - $C_{18}$  alkyl group or the corresponding ester linkage interrupted group with a short alkylene ( $C_1$ - $C_4$ ) group between the ester linkage and the N, and having a similar

- 10 -

hydrocarbon group, e.g., a fatty acid ester of choline, preferably C<sub>12</sub>-C<sub>14</sub> (coco) choline ester and/or C<sub>16</sub>-C<sub>18</sub> tallow choline ester are used. Each R is a C<sub>1</sub>-C<sub>4</sub> alkyl or substituted (e.g. hydroxy) alkyl, or hydrogen, preferably methyl, and the counterion X<sup>-</sup> is a compatible anion, for example, chloride, bromide, methyl sulphate etc. Such surfactants can protect the quaternary ammonium conditioning compound from interacting with anionic surfactants and/or detergent builders that are carried over into the rinse.

10

The stabilising agent may be a mixture selected from different nonionic surfactants and/or single long chain alkyl cationic surfactants.

15 The compositions of the invention also contain a fatty alcohol.

Suitable fatty alcohols which can be used in the present invention include fatty alcohols of about 10 to 40 carbon atoms. More preferably the fatty alcohol may have a straight or branched chain alkyl or alkenyl group having from about 10 to 24, preferably from about 10 to 20, especially from about 12 to 20 carbon atoms. Specific examples of the fatty alcohol include decanol, dodecanol, tetradecanol, pentadecanol, hexadecanol, octadecanol, and mixtures thereof.

20  
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- Furthermore, the fatty alcohol may be of a natural or synthetic origin and may include, for example, mixed alcohol, such as C<sub>16</sub> to C<sub>18</sub> alcohols prepared by Ziegler polymerisation of ethylene or by reduction of fatty acids.
- 5 The fatty alcohol is preferably present in the composition at a ratio equal to or greater than 1:1 relative to the stabilising agent. Preferably, it is present in an amount of 0.1 to 20% by weight of the composition, most preferably 0.3% to 12%, more preferably 0.5% to 10%, such as 0.5% to
- 10 5%. Frequently the compositions of the invention may comprise of 0.1 to 2% by weight, such as 0.1 to 1% of the fatty alcohol.
- We have found that particularly good compositions of the
- 15 invention comprise 1 to 8% by weight of the quaternary ammonium compound, from 0.01 to 5% by weight of the nonionic stabilising material and between 0.2 to 20% by weight of the fatty alcohol.
- 20 The composition can also contain one or more optional ingredients, selected from non-aqueous solvents, pH buffering agents, perfumes, perfume carriers, fluorescers colourants, hydrotopes, antifoaming agents, antirededposition agents, enzymes, optical brightening agents, opacifiers,
- 25 anti-shrinking agents, anti-wrinkle agents, anti-spotting agents, germicides, fungicides, anti-oxidants, anti-corrosion agents, drape imparting agents, anti-static agents and ironing aids.

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The composition may also contain nonionic fabric softening agents such as lanolin and derivatives thereof.

The compositions of the invention may be prepared by a  
5 method where the cationic fabric softening agent,  
stabilising agent and fatty alcohol are premixed and heated  
together to form a clear melt. The molten mixture thus  
formed is added to water at a suitable temperature to keep  
the aforementioned components molten, e.g. 70°C to 80°C,  
10 over a period of at least one minute, with constant stirring  
to form a dispersion.

The compositions of the invention may be used to form stable  
aqueous fabric softening compositions. Also according to  
15 the invention in fabric softening compositions comprising 1  
to 8% by weight of one or more quaternary ammonium fabric  
conditioning compounds, a mixture of stabilising agent and  
fatty alcohol in a weight ratio of greater than or equal to  
1:1 is used to improve the stability of the composition at  
20 low, ambient and high temperatures.

Preferably, according to these aspects of the method of  
preparing the compositions and their uses, the cationic  
fabric softening agent is represented by one of the two  
25 formulae set out above. Other preferred features of the  
use, in particular with respect to the non-ionic stabiliser  
and fatty alcohol, correspond to those for the first aspect  
of the invention.

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The invention will now be illustrated by the following non-limiting examples. In the examples all percentages are expressed by weight.

## 5 Examples

Liquid fabric softening compositions were made as follows.

The cationic fabric softening agent, stabilising agent and fatty alcohol were premixed and heated together to form a clear melt. The molten mixture thus formed was added to water at 70°C to 80°C over a period of at least one minute, with constant stirring to form a dispersion.

Composition	A	B <sup>7</sup>	C	D	E <sup>7</sup>	F	G
DEEDMAC <sup>1</sup>	4.8	4.8	4.6	4.4	4.8	4.6	4.4
Genapol C200 <sup>2</sup>	0.1	0.1	0.1	0.1	0.2	0.2	0.2
Laurex CS <sup>3</sup>	0.75		0.36	0.72		0.36	0.72
Perfume <sup>4</sup>	0.32						
Natrasol plus 331 <sup>5</sup>	.005						
Water and minors <sup>6</sup>	----- up to 100% -----						

## 15 NOTES

1. DEEDMAC is a Di[2-(hardened tallowoxyloxy)ethyl] dimethylammonium chloride.
- 20 2. Coco alcohol ethoxylated with 20 moles of ethylene oxide ex Hoechst AG.
3. Tallow alcohol ex Albright & Wilson.

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4. Softline DM53 ex Givaudan-Rouve
5. Thickener/polymer.
- 5 6. Minors associated with the raw materials present in all components of the examples.
7. Examples B and E are comparative as they do not contain any fatty alcohol.

10

The viscosity of the compositions A - G was measured by Haake rotoviscometer after 1, 4 and 6-8 weeks storage at ambient temperature or at 37°C. The results below were obtained.

15

Viscosity at 110s <sup>-1</sup> mPas	A	B	C	D	E	F	G
Initial	70	130	60	105	23	45	83
1 week at 37°	63	79	41	90	12	40	71
2	58	63	39	85	11	38	71
3	57	52	41	88	9	36	66
4	55	30	35	75	4	27	67
5	55	20	34	83	5	34	83
6	55	15	32	79	5	33	73
7		15	32	79	5	37	77
8	56	10	32	71	4	35	76
Initial	70						
1 week at ambient	61	130	60	105	23	45	83
2	56	110	43	87	21	35	67
3	58	110	44	85	21	38	63



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4	54	106	45	90	20	38	70
5	56	102	44	81	12	33	72
6	53	102	44	81	12	33	72
7		98	47	89	10	38	63
8	56	87	44	75	7	32	63

By comparison with comparative examples B and E these results show that addition of a mixture of fatty alcohol and nonionic stabiliser to compositions comprising ester linked

5 quaternary ammonium compounds counteracts destabilisation at ambient and high temperatures to give more stable compositions.

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CLAIMS

1. A fabric softening composition comprising:

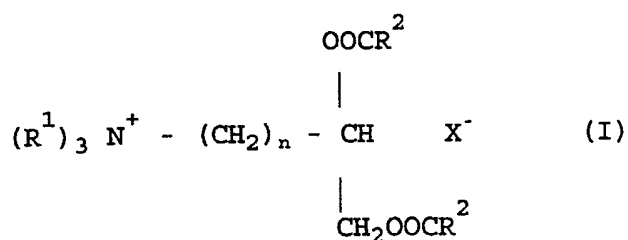
- 5           a)    from 1 to 8% by weight of one or more quaternary ammonium fabric conditioning compounds;
- b)    a stabilising agent selected from a nonionic surfactant or a single long chain alkyl cationic surfactant or mixtures thereof ;and
- 10           c)    a fatty alcohol.

2. A composition according to claim 1 wherein one or more of the one or more quaternary ammonium fabric conditioning compounds has at least one ester linkage.

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3. A composition according to either claim 1 or claim 2 in which one or more of the quaternary ammonium fabric conditioning compounds is represented by the formula:

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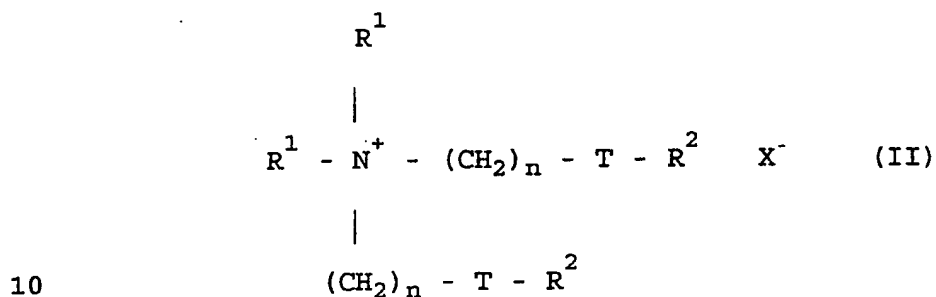
wherein each  $\text{R}^1$  group is independently selected from  $\text{C}_1$ - $\text{C}_4$  alkyl, alkenyl or hydroxyalkyl groups; each  $\text{R}^2$  group is independently selected from  $\text{C}_8$ - $\text{C}_{28}$  alkyl or alkenyl

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groups;  $X^-$  is a compatible anion; and  $n$  is an integer from 0-5;

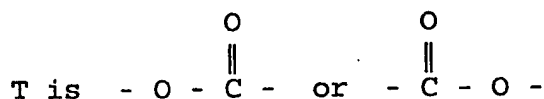
or by the formula:

5



wherein  $R^1$ ,  $X^-$ ,  $n$  and  $R^2$  are as defined above, and

15



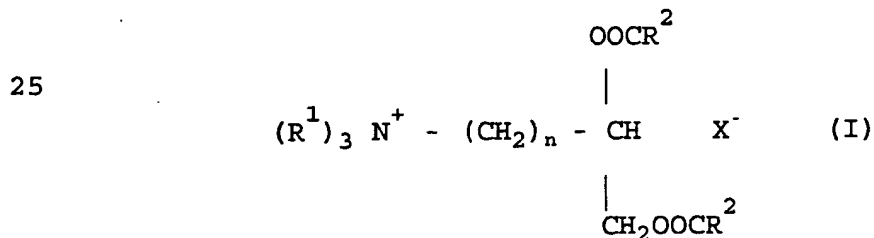
4. A composition according to anyone of claims 1 to 3 wherein the quaternary ammonium compound is 1,2 bis[hardened tallowoyloxy]-3-trimethylammonium propane chloride or di[2-(hardened tallowoyloxy)ethyl] dimethylammonium chloride.
- 20
5. A composition according to claim 1 wherein the quaternary ammonium compound is di-(hardened tallow) dimethylammonium chloride.
- 25

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6. A composition according to any preceding claim wherein the weight ratio of b:c is greater than or equal to 1:1.
- 5 7. A composition according to any preceding claim wherein the stabilising agent comprises a C<sub>8</sub>-C<sub>30</sub> primary or secondary linear or branched alcohol alkyoxylated with 10 or more moles of alkylene oxide.
- 10 8. A composition according to any preceding claim wherein the fatty alcohol comprises a chain length of about 10 to 40 carbon atoms.
- 15 9. A composition as claimed in any preceding claim wherein the composition comprises 1 to 8% by weight of the quaternary ammonium compound, from 0.01 to 5% by weight of the nonionic stabilising material and between 0.2 to 20% by weight of the fatty alcohol.
- 20 10. A process for making a liquid fabric softening composition as claimed in any of claims 1 to 9 comprising the steps of
- 25 (i) mixing and heating the quaternary ammonium compound, stabilising agent and fatty alcohol to form a melt and
- (ii) dispersing the melt in water.

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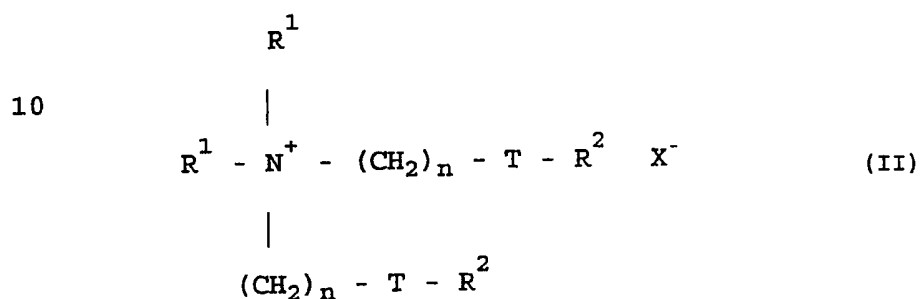
11. Use of a composition as defined in any of claims 1-9 to form a stable aqueous fabric softening composition.
12. Use, in a fabric softening composition comprising 1-8% by weight of one or more water insoluble quaternary ammonium compounds, of a mixture of a stabilising agent and fatty alcohol, in order to improve the low, ambient and high temperature stability of the composition.
13. Use, according to either of claims 11 or 12 wherein the ratio of stabilising agent and fatty alcohol is greater than or equal to 1:1.
14. Use, according to either claim 12 or 13, wherein the stabilising agent is the condensation product of C<sub>8</sub>-C<sub>30</sub> primary or secondary linear or branched alcohols alkoxyated with 10 or more moles of alkylene oxide and wherein the fatty alcohol has between 10 and 40 carbon atoms.
15. Use according to any of claims 11-14 wherein the quaternary ammonium compound is represented by the formula:



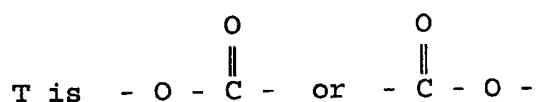
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wherein each  $R^1$  group is independently selected from  $C_{1-4}$  alkyl, alkenyl or hydroxyalkyl groups; each  $R^2$  group is independently selected from  $C_{8-28}$  alkyl or alkenyl groups;  $X^-$  is a compatible anion; and  $n$  is an integer from 0-5;

or by the formula:



wherein  $R^1$ ,  $n$ ,  $X^-$ , and  $R^2$  are as defined above, and



# INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 99/01563

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C11D3/00 C11D3/20 C11D1/645 C11D1/835

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 94 04643 A (COLGATE PALMOLIVE CO) 3 March 1994 see claims 1-4,8; example 1B ---	1-3,7-9
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Date of the actual completion of the international search

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International Application No

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